

Tetraphenylborate Method

Method 10321
0.1 to 7.0, 1.0 to 70, 10 to 700 mg/L K
Powder Pillows
Scope and application: For oil and gas field waters.

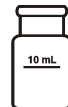
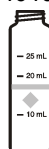

Test preparation

Instrument-specific information

[Table 1](#) shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	
DR900	The orientation mark is toward the user.	2401906 

Before starting

For turbidimetric methods, install the instrument cap or cover on all instruments before ZERO or READ is pushed.

Clean sample cells with soap, water and a brush soon after each test to prevent a build-up of film on the sample cells.

Filter samples that are turbid with filter paper and a funnel.

The test results can vary with different lots of reagent. For best results, calibrate the instrument with each new lot of reagent. Refer to [Calibration](#) on page 5.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Description	Quantity
Sodium Gluconate Powder Pillow	1
Lithium Hydroxide Powder Pillow	1

Items to collect (continued)

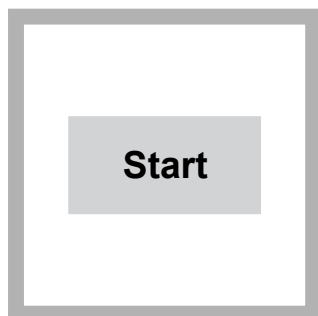
Description	Quantity
Potassium 4 Reagent Powder Pillow	1
Mixing cylinder, graduated, 25 mL, glass stopper	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 6 for order information.

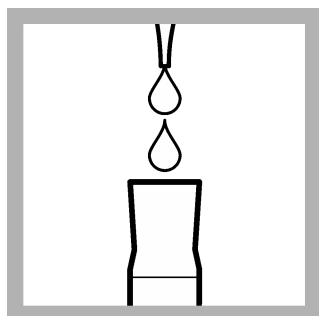
Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 4–5 with 5 N sodium hydroxide solution.
- A pH probe can contaminate the sample. If a pH probe is used, pour a portion of sample into a separate beaker for pH measurement or use pH paper.
- Correct the test result for the dilution caused by the volume additions.

Test procedure



1. Start program 905 Potassium. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



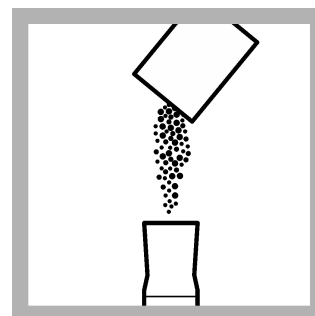
2. Prepare the sample: Add the sample volume that is specified for the test range to a 25-mL mixing cylinder:

- 0.1–7.0 mg/L: 25 mL
- 1.0–70 mg/L: 2.5 mL
- 10–700 mg/L: 0.25 mL

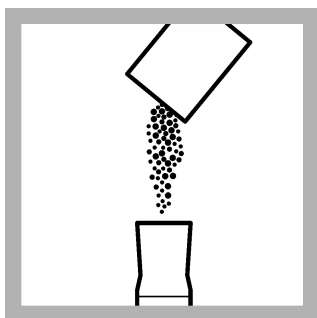
Use a pipet to measure 0.25 mL or 2.5 mL.



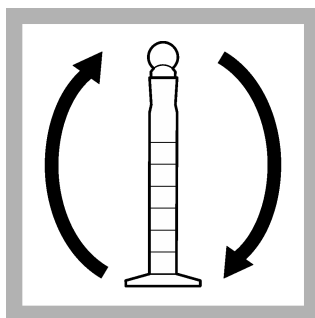
3. If the sample volume is less than 25-mL, add deionized water to the 25-mL line.



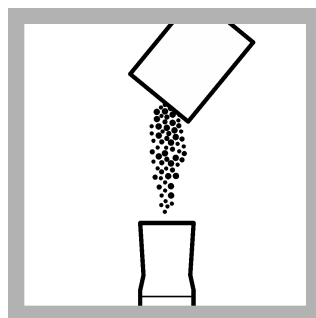
4. Add the contents of one Sodium Gluconate Pillow.



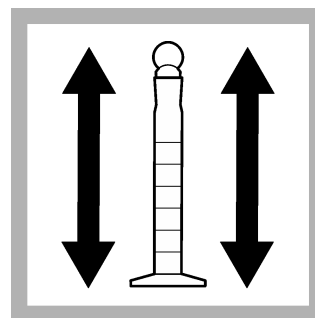
5. Add the contents of one Lithium Hydroxide Pillow.



6. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix. Let the solution become clear.



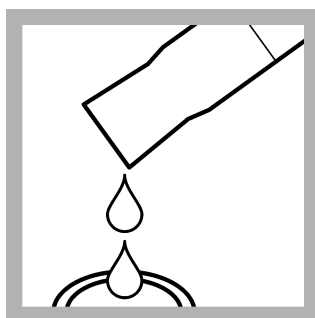
7. Add the contents of one Potassium 4 Reagent Pillow.



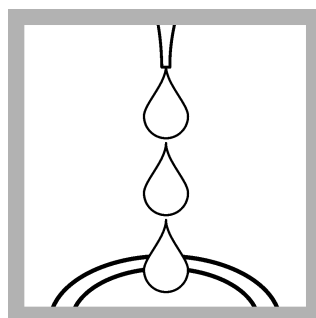
8. Put the stopper on the mixing cylinder. Shake the cylinder for 30 seconds. A white turbidity forms if potassium is in the sample.



9. Start the instrument timer. A 3-minute reaction time starts.



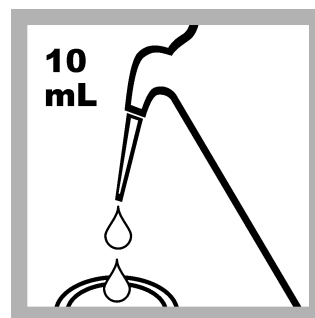
10. Pour 10 mL of the solution from the mixing cylinder into the sample cell.



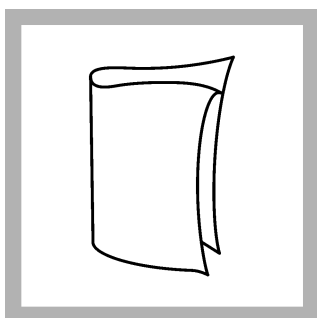
11. **Prepare the blank:** Add the untreated sample volume that is specified for the test range to a second 10-mL sample cell:

- 0.1–7.0 mg/L: 10 mL
- 1.0–70 mg/L: 1.0 mL
- 10–700 mg/L: 0.1 mL

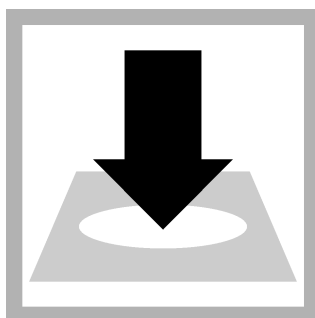
Use a pipet to measure 0.1 mL or 1.0 mL.



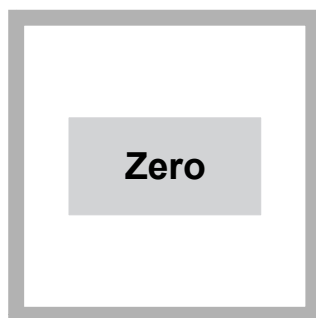
12. If the sample volume is less than 10-mL, add deionized water to the 10-mL line.



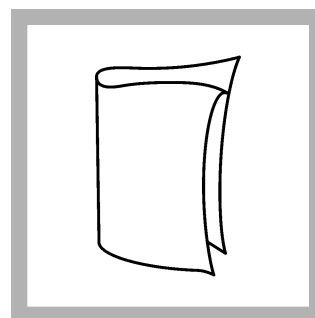
13. When the timer expires, clean the blank sample cell.



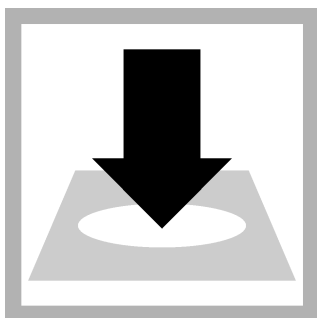
14. Insert the blank into the cell holder.



15. Push **ZERO**. The display shows 0.0 mg/L K.



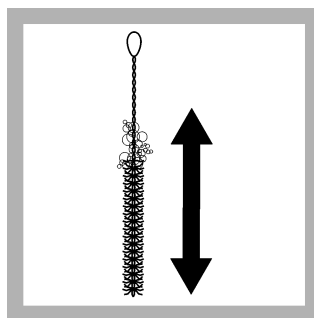
16. Clean the prepared sample cell.



17. Within 7 minutes after the timer expires, insert the prepared sample into the cell holder.



18. Push **READ**. Results show in mg/L K.



19. Immediately clean the graduated cylinder and sample cells with soapy water and a brush. Rinse with deionized water.

Interferences

Table 2 shows the substances that were tested in non-diluted samples and will not interfere at or below the levels stated. If these substances are present at higher levels, conduct interference studies at the higher levels to determine if the substance interferes.

Table 2 Interfering substances

Interfering substance	Interference level
$\text{NH}_4^+ - \text{N}$	15 mg/L
Ca^{2+} as CaCO_3	7000 mg/L
Cl^-	15,000 mg/L
Mg^{2+} as CaCO_3	6000 mg/L

Set the dilution factor

Instruments that have a dilution factor option can include the dilution factor in the result and show the concentration of the original, undiluted sample. For example, if the sample is diluted by a factor of 10, the instrument multiplies the result by 10 and shows the calculated result in the instrument display.

1. Select **Options>More>Dilution** factor from the instrument menu.
Note: DR1900: Select **Options>Advanced Options>Dilution Factors>On**.
Note: Colorimeters include a dilution factor when the chemical form is set. Go to **Options>Advanced Options>Chemical Form** and select LR, MR or HR.
2. Enter the dilution factor:
 - 1 mL sample diluted to 10 mL: dilution factor is 10.
 - 0.1 mL sample diluted to 10 mL: dilution factor is 100.
3. Push **OK** to confirm. Push **OK** again.
4. Push **RETURN** to go back to the measurement screen.

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Note: This procedure is not applicable to user programs.

Items to collect:

- 250-mg/L Potassium Standard Solution
- 25-mL mixing cylinders, graduated (3)

- Pipet, TenSette®, 0.1–1.0 mL and tips
 - Ampule breaker
1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
 2. Go to the Standard Additions option in the instrument menu.
 3. Select the values for standard concentration, sample volume and spike volumes.
 4. Open the standard solution.
 5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 25-mL portions of fresh sample. Mix well.
 6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
 7. Select **Graph** to compare the expected results to the actual results.

Note: If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.

Calibration

This method requires a user-prepared calibration curve. Enter the calibration curve into the instrument as a user program. Make a new calibration curve when a new lot of reagents is used.

Prepare the standard solutions

Prepare the standard solutions for calibration as follows.

Items to collect:

- 100-mg/L Potassium Standard Solution
 - 100-mL volumetric flasks (8), Class A
 - 1–10 mL TenSette pipet and tips
 - Deionized water
1. Prepare eight calibration standard solutions (1, 2, 3, 4, 5, 6, 7 and 8 mg/L potassium) as follows:
 - a. Use a pipet to add 1, 2, 3, 4, 5, 6, 7 and 8 mL of the 100-mg/L standard solution into eight different 100-mL volumetric flasks.
 - b. Dilute each flask to the mark with deionized water. Mix well.
 2. Use deionized water for the 0 mg/L potassium standard.
 3. Go to user programs and enter the calibration information. Refer to [Enter the calibration as a user program](#) on page 5.

Enter the calibration as a user program

After the calibration standards are prepared, make a user program to store the calibration information in the instrument. Select the user program to measure the concentration of samples. The steps that follow are general instructions for all instruments. Refer to the user manual for the instrument that is used for the correct menu options.

1. Go to **User Programs**.
2. For the initial calibration, make a new user program. Set up the basic information for the new program:

Option	Description
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Program number	Enter an available number for the user program.
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Option	Description
Program name	Enter a name for the user program, (e.g., the name of the parameter).
Program type	Select single wavelength (for applicable spectrophotometers).

3. Enter the settings for the user program:

Option	Description
Units	mg/L
Wavelength	650 nm for spectrophotometers or 610 nm for colorimeters
Concentration resolution	0.1
Chemical form	K
Upper limit	8.0
Lower limit	−0.2
Timer 1	3:00
Calibration	Read standards

4. In the Read Standards menu, enter the concentration of the prepared standard solutions.
5. Use the test procedure to prepare the standard solutions for measurement.
6. Insert the blank solution into the cell holder and push **ZERO**.
7. Insert the first prepared standard solution into the cell holder. Make sure that the standard solution concentration is selected on the display and push **READ**.
8. Continue to measure the remaining standard solutions.
9. When all of the standard solutions have been measured, compare the graph options. Select the best curve.
10. Save (store) the user program. When the user program is selected in the test procedure, the calibration curve is used to measure the sample concentration.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
905	5.0 mg/L K	4.7–5.3 mg/L K	0.1 mg/L K

Summary of method

Potassium in the sample reacts with sodium tetraphenylborate to form potassium tetraphenylborate, an insoluble white solid. The amount of turbidity produced is proportional to the potassium concentration. The measurement wavelength is 650 nm for spectrophotometers or 610 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Potassium Reagent Set, includes:	—	—	2460000
Sodium Gluconate Powder Pillow	1	100/pkg	2590599
Lithium Hydroxide Powder Pillow	1	100/pkg	1416369
Potassium Reagent 4 Powder Pillow	1	100/pkg	1432399NF

Required apparatus

Description	Quantity/test	Unit	Item no.
Mixing cylinder, graduated, 25 mL, glass stopper	1	each	189640
Pipet, adjustable volume, 1.0–5.0 mL	1	each	BBP065
Pipet tips, for 1.0–5.0 mL pipet	1	75/pkg	BBP068
Pipet, adjustable volume, 0.1–1.0 mL	1	each	BBP078
Pipet tips, for 0.1–1.0 mL pipet	2	100/pkg	BBP079

Recommended standards

Description	Unit	Item no.
Potassium Standard Solution, 10-mL Voluette [®] Ampule, 250 mg/L	16/pkg	1479010
Potassium Standard Solution, 100-mg/L	500 mL	2351749

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette [®] Ampules	each	2196800
Brush, test tube	each	69000
Flask, volumetric, Class A, 100 mL	each	1457442
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Nitric Acid, concentrated	500 mL	15249
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet, TenSette [®] , 1.0–10.0 mL	each	1970010
Pipet tips for TenSette [®] Pipet, 1.0–10.0 mL	50/pkg	2199796
Sodium Hydroxide Solution, 5 N	50 mL	245026
Water, deionized	4 L	27256



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